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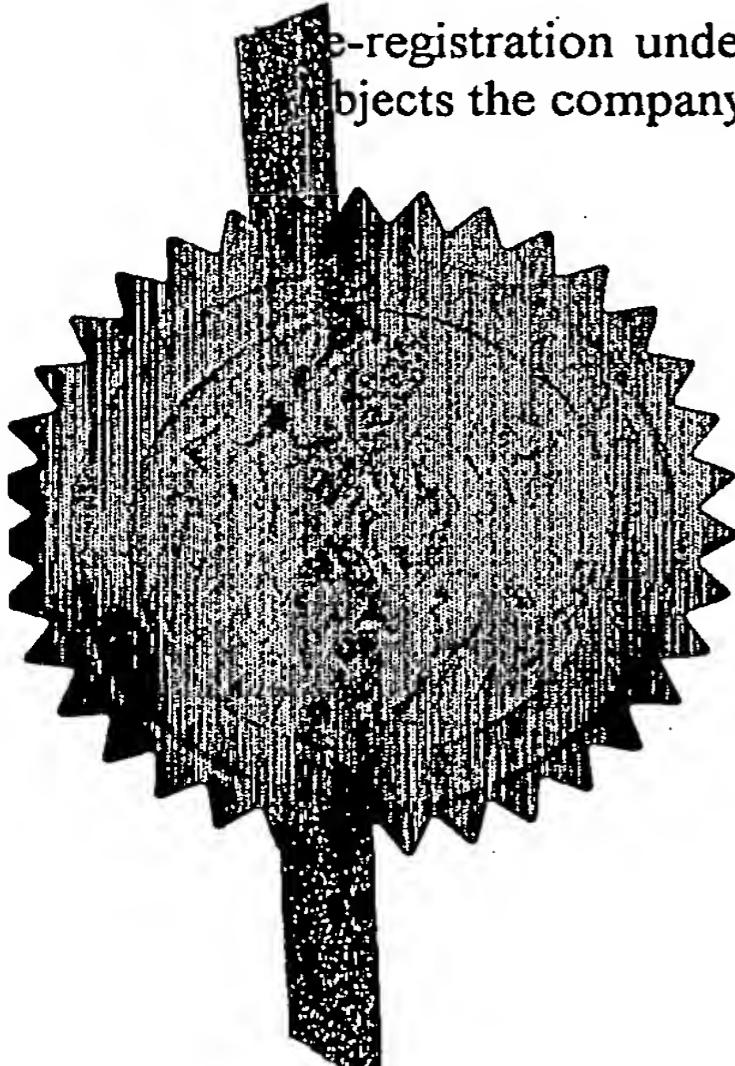
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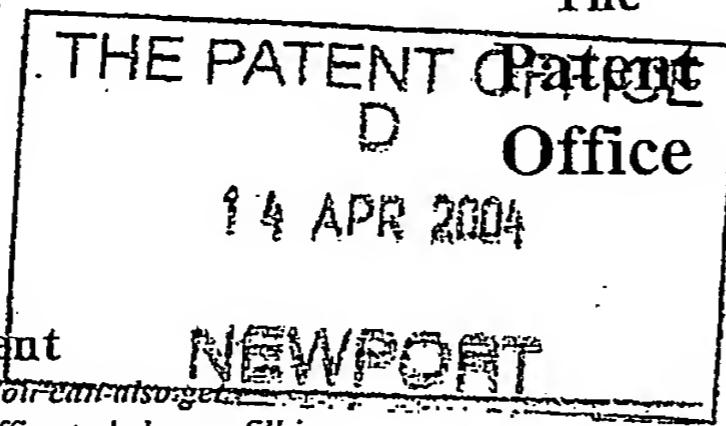
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14APR04 E866294-2 D02898  
P01/7700 0.00-0408249.1 ACCOUNT CHA

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1. Your reference

F2044 (C)/ps

0408249.1

14 APR 2004

2. Patent application number

(The Patent Office will fill this part in)

3. Full name, address and postcode of the or of each applicant (*underline all surnames*)

UNILEVER PLC  
UNILEVER HOUSE, BLACKFRIARS  
LONDON, EC4P 4BQ

Patents ADP number (*if you know it*)

-50426956002 1628002

If the applicant is a corporate body, give the country/state of its incorporation

UNITED KINGDOM

4. Title of the invention

IMPROVED PROCESS FOR TEA MANUFACTURE

5. Name of your agent (*if you have one*)

ELLIOTT, Peter William

"Address for Service" in the United Kingdom to which all correspondence should be sent (*including the postcode*)

PATENT DEPARTMENT, UNILEVER PLC  
COLWORTH HOUSE, SHARNBROOK  
BEDFORD, MK44 1LQ

Patents ADP number (*if you know it*)

1628003

6. Priority: Complete this section if you are declaring priority from one or more earlier patent applications, filed in the last 12 months.

Country	Priority application number ( <i>if you know it</i> )	Date of filing ( <i>day / month / year</i> )
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7. Divisionals, etc: Complete this section only if this application is a divisional application or resulted from an entitlement dispute (see note f)

Number of earlier application	Date of filing ( <i>day / month / year</i> )
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8. Is a Patents Form 7/77 (Statement of inventorship and of right to grant of a patent) required in support of this request?

YES

Answer YES if:

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
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Patents Form 1/77

9. Accompanying documents: A patent application must include a description of the invention. Not counting duplicates, please enter the number of pages of each item accompanying this form:

Continuation sheets of this form

Description	14
Claim(s)	3
Abstract	1
Drawing(s)	-

10. If you are also filing any of the following, state how many against each item.

Priority Documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

1

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature(s)

Date: 13/04/04

Sandra Jane EDWARDS, Authorised Signatory

12. Name, daytime telephone number and e-mail address, if any, of person to contact in the United Kingdom

Petra Silverstone (01234) 222893  
petra.silverstone@unilever.com

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Improved Process for Tea ManufactureField of the Invention

This invention relates to a process for obtaining cold water infusible or extractable tea starting with black tea as the raw material and to the products obtained thereby that have improved red colour, infuse faster and have good flavour.

Background of the Invention

Leaf tea may be prepared as green leaf tea or black leaf tea. Generally, to prepare black leaf tea, fresh green leaves of the plant *Camellia sinensis* are withered (a process to allow the plucked tea leaves to lose moisture and bring about chemical / biochemical changes especially in aroma), macerated, fermented (in which process enzymes in the tea leaf use atmospheric oxygen to oxidise various substrates to produce coloured products) and then dried at higher temperatures (to stop the enzyme activities). Whereas, green tea is not exposed to the fermentation process and partial fermentation may be used to produce intermediate-type teas known as "oolong" tea.

Tea is consumed as a hot beverage or as a cold beverage (for example iced tea). The numerous compounds in the leaves that give the beverage its unique organoleptic properties are only sparingly soluble in cold water therefore tea is usually infused in water at temperatures close to 100°C.

Cold water soluble teas are usually prepared by spray drying the liquor obtained by extraction of black tea or fibres generated during the black tea manufacturing process. However, this process requires high temperatures or treatment with harsh chemicals like alkalis, which adversely affect the tea attributes like taste, colour and flavour.

There have been many methods reported to treat green tea to prepare tea products that infuse in cold water. US 4,051,264 (Lipton/Sanderson) describes a process that involves treating green tea leaves with enzymes during the processing of tea to 5 generate cold water infusing teas. US 3,812,266 (Sanderson/Coggon) discloses a method that involves converting green tea to black tea using tannase and natural tea enzymes to convert green tea into black, and generate tea powders, which are both hot and cold water infusible.

10 Apart from the advantages of obtaining cold water infusible tea, an important consideration for the consumer is tea colour, brightness and aroma. Tea colour refers to the colour of the infusion with or without milk. Black tea infusions can range from yellow to red-brown in colour. Teas with bright, red 15 liquor and good aroma are particularly preferred in countries like India and are perceived to be 'strong' teas. It is thus desirable to produce tea with these characteristics. Moreover, it is desirable to produce tea that infuses faster in water as these teas are perceived to be strong teas. Thus teas 20 that infuse fast and provide a good red colour are much preferred by consumers and are perceived to be teas with good strength.

Many methods of treating green tea to produce tea products that give bright red colour liquor in water have been reported. US 25 5863581 (Lipton, Division of Conopco, Inc.) discloses a process for manufacturing a tea product where zeolites are used to generate red coloured teas. Tadao Kurata et al in Agr. Biol. Chem., 37 (6), 1471-1477, 1973 discloses that a red pigment is produced at the initial stage of the browning reaction of 30 dehydro-L-ascorbic acid (DHA) with alpha-amino acid. 5-phenyl-3,4-diketo-gamma-butyrolactone, which has the same type of lactone ring structure as dehydro-L-ascorbic acid, is said to give a similar red colour when reacted with alpha-amino acid.

The pigment is said to have the same structure as the red pigment that is produced by the oxidation of L-scorbamic acid.

WO 01/70038 (Unilever, 2001) concerns a process for manufacturing a cold water infusible black leaf tea comprising 5 macerating freshly plucked tea leaves, allowing them to ferment, firing the leaves to arrest fermentation and then drying them to yield black leaf tea. The process is characterised in that the tea leaves are treated with a solubilising compound selected from the group consisting of 10 ascorbic acid, dehydroascorbic acid, L-scorbamic acid or 5-phenyl-3,4-diketo-gamma-butyrolactone, preferably during the maceration step. The black leaf tea so produced is soluble in water at 5 to 100°C. The teas have a good red colour. The publication also teaches that the tea leaves are preferably 15 treated with the solubilising compound in the presence of oxidative enzymes and/or hydrogen peroxide to enhance the tea flavour and colour.

JP 47-49719 (Tanabe, 1972) reports a method of improving the taste and flavour of tea during its manufacture by adding amino 20 acids and 1,3-dihydroxy-2-propanone at an appropriate heating stage.

GB 2348 104 (Unilever, 2000) reports a method of manufacturing black tea with improved aroma comprising the step of treating green leaf tea with a combination of phenolic acids and amino 25 acids prior to fermentation step.

JP 02128669 (Ajinomoto, 1990) reports a method to improve flavour and reduce bitter taste of tea by adding amino acid wherein amino acid and tea are in the range of 0.1 to 60% and 0.1 to 50% respectively.

All of the above methods have been reported to treat green tea during the process of manufacture of black tea to prepare cold water infusible tea or enhancing other attributes such as flavour & bitterness reduction. There has been a need to 5 provide for cold water infusible teas that has good red colour and more importantly fast infusion rate in water starting with black tea as the raw material. This is especially useful in countries where there are no tea gardens and there are no tea processing factories. Black tea is a comparatively more stable 10 product and so black tea can be imported and the process of making this fast infusing cold water infusible tea can be carried out at location far remote from the tea gardens. The present inventors have now found that one can prepare a tea product that provides a high quality tea beverage when infused 15 in hot or cold water and provides a rich bright red colour infusion in fast time by treating black tea with ascorbic acid or its salts/derivatives, one or more amino acids and an oxidising agent.

#### Objects of the Invention

20 It is thus an object of the invention to provide for a cold or hot water infusible tea product with improved red colour and good flavour.

25 It is a further object of the invention is to provide for a cold or hot water infusible tea that infuses quickly into the water.

#### Definition of the Invention

According to the first aspect of the invention, there is provided a 30 process for manufacturing a tea product comprising contacting black tea with ascorbic acid its salts or mixtures thereof, one or more amino acids, an oxidizing agent and water for a period of at least 5

minutes followed by drying to prepare a tea product that is infusible in water at 5 to 100°C.

According to a preferred embodiment of the invention, there is provided a process for manufacturing a black tea product comprising 5 contacting black tea with amounts of, by weight of black tea 1 to 8% ascorbic acid its salts or mixtures thereof; 0.1 to 8% of one or more amino acids and 0.3 to 7% of an oxidizing agent for a period of at least 5 minutes followed by drying to less than 10 5% moisture to prepare a black tea that is infusible in water at 5 to 100°C. Wherein the ascorbic acid its salts or mixtures thereof, the one or more amino acids and the oxidising agent are added as aqueous solutions.

According to a further preferred embodiment of the invention, there 15 is provided a process for manufacturing a tea product comprising of contacting black tea with amounts of, by weight of black tea 2 to 4% ascorbic acid its salts or mixtures thereof, 0.1 to 2% of one or more amino acids and 0.3 to 5% of hydrogen peroxide 20 for a period of 5 minutes to 24 hours followed by drying to less than 5% moisture to prepare a tea product that is infusible in water at 5 to 100°C. Wherein the ascorbic acid its salts or mixtures thereof, the one or more amino acids and the oxidising agent are added as aqueous solutions.

25 The invention also pertains to the tea obtained or obtainable by this process. The tea so obtained is cold water and hot water infusible/extractable and the infusion/extract shows improved red colour. The tea also infuses quickly into the water.

It is particularly preferred that the black tea is first contacted 30 with the ascorbic acid its salts or mixtures thereof before it is contacted with the oxidising agent.

Detailed Description of the Invention

"Tea" for the purposes of the present invention means leaf material from *Camellia sinensis* var. *sinensis* or *Camellia sinensis* var.

5 *assamica*. It also includes rooibos tea obtained from *Aspalathus linearis* however that is a poor source of endogenous fermenting enzymes. "Tea" is also intended to include the product of blending two or more of any of these teas.

10 "Leaf tea" for the purposes of this invention means a tea product that contains one or more tea origins in an uninfused form.

"Cold water infusible" for the purposes of this invention means giving good colour, flavour and mouthfeel in a short infusion time 15 i.e. less than 10 minutes, but preferably less than 5 minutes at a temperature at or between 5 and 30°C.

For the avoidance of doubt the word "comprising" is intended to mean including but not necessarily "consisting of" or "composed of". In 20 other words the listed steps or options need not be exhaustive.

Tea manufacture, especially black tea manufacture, traditionally comprises: withering, macerating, fermenting and firing. Black tea for the purpose of the invention is obtained by the above process or 25 by any other known process to prepare black tea.

The present invention concerns further treatment of the black tea obtained as described above to prepare a cold water infusible tea product. The treatment involves contacting black tea (leaf /dust) or 30 secondaries or green leaf mixed black tea or off graded black tea or tea solids coated black tea with ascorbic acid its salts or mixtures thereof, one or more amino acids, and an oxidising agent preferably hydrogen peroxide with or without peroxidase addition, in the presence of moisture, in order to enhance the infusion of the black

tea in cold water. All these agents are preferably added in the form of aqueous solutions. Alternately they may be dry mixed followed by addition of water. The tea is thus contacted for at least 5 minutes and preferably dried to less than 5% moisture. The solution can be 5 applied singly or in split doses. The treatment is preferably given in the form of a spray or dip. The black tea can be treated either ex-drier mouth or after grading.

10 Ascorbic acid its salts or mixtures thereof including sodium or calcium salts are preferably added in an amount ranging from 1% to 8% by weight of tea, more preferably in an amount ranging from 2% to 4% by weight of tea. The ascorbic acidits salts or mixtures thereof are preferably added as an aqueous solution.

15 Amino acid can be used singly or in combination. Amino acids as per this invention include, alanine, arginine, asparagin, cystine, glutamine, glycine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, serine, threonine, tryptophan, tyrosine and valine. They are either mixed with ascorbic acid or can be added 20 separately and the amount is preferably between 0.1% to 8% by weight of tea, more preferably 0.1% to 2% by weight of tea. The resultant solution of ascorbic and amino acid can be applied singly or in split doses. The treatment is preferably given in the form of a spray or dip.

25 Preferably, aqueous solutions of ascorbic acid its salts or mixtures thereof and amino acids should be heated to a temperature of at least 50°C for contacting with tea. The amount of water can be between 10% to 100% by weight of tea, more preferably between 30% to 30 100% by weight of tea, most preferably between 40% to 100%.

It is preferred that the oxidising agent is added to the black tea after the ascorbic acids and/or its salts and the amino acids are added. The oxidising agent may be hydrogen peroxide, calcium

peroxide, magnesium peroxide, oxygen or any compound, which can release oxygen under the process conditions, or may be an enzyme which can release hydrogen peroxide. Preferred oxidising agent is hydrogen peroxide. The oxidising agent is preferably added in an 5 amount of 0.3% to 7% by weight of tea. When the oxidising agent is hydrogen peroxide it may be added as a 10% to 75% solution in water. Usually hydrogen peroxide is available at 30% hydrogen peroxide solution in water. The oxidising agent is more preferably added in an amount of from 0.3% to 5% by weight of tea. The solution 10 can be applied singly or in split doses. The treatment is preferably given in the form of a spray or dip.

The ascorbic acid, amino acids, water and oxidising agent are preferably kept contacted with black tea, a process step known as 15 incubation for a period of at least 5 minutes. The incubation may be carried out for a period of up to 24 hours. The incubation is preferably carried out at a temperature of 10 to 60°C.

The tea can be dried using any method/process known in the art, 20 preferably giving tea with a moisture content of less than 5%.

Water infusions of the tea produced by the process described above in water at temperatures in the range of 5 to 100°C will give tea with superior colour and flavour.

25 It will be appreciated that the process of the invention can be advantageously used for improving the quality of and adding value to low grade black teas without wastage.

30 The process of the invention shall now be described with reference to the following non-limiting examples:

Examples**Example 1:**

Ascorbic acid (3 g) and phenylalanine (0.5 g) were dissolved in  
5 100 mL of hot water. This solution was further heated on a  
boiling water bath for 5 minutes. The hot solution was sprayed  
on 100 grams of black tea. About 5 mL of 30% hydrogen peroxide  
solution was added to it. This was incubated at 25 - 30°C for  
about 15 minutes followed by drying on a fluidised bed drier at  
10 130 - 140°C to bring down the moisture to less than 5% on black  
tea basis.

**Comparative Example A:**

100 grams of black tea was wetted with 100 mL hot water. This  
15 was incubated around 25 - 30°C for about 15 minutes followed by  
drying on a fluidised bed drier at 130 - 140°C to bring down  
the moisture to less than 5% on black tea basis.

**Comparative Example B:**

20 A process as per Comparative Example A was carried out except  
that additionally 5 ml of 30% hydrogen peroxide was sprayed  
immediately after the addition of water.

**Comparative Example C:**

25 A process as per Comparative Example A was carried out except  
that a solution of 0.5 g of phenylalanine in 100 mL water,  
which had been heated on a boiling water bath for 5 minutes,  
was sprayed on the tea instead of the 100 mL of water used in  
Comparative Example A.

30

**Comparative Example D:**

A process as per Comparative Example C was carried out except  
that 3 g of ascorbic acid was used in place of 0.5 g of  
phenylalanine.

**Comparative Example E:**

Ascorbic acid (3 g) was dissolved in 100 mL of hot water. This solution was further heated on a boiling water bath for 5 minutes. The hot solution was sprayed on 100 grams of black tea. About 5 mL of 30% hydrogen peroxide solution was added to it. This was incubated at 25 - 30°C for about 15 minutes followed by drying on a fluidised bed drier at 130 - 140°C to bring down the moisture to less than 5% on black tea basis.

10

**Comparative Example F:**

A process as per Comparative Example E was carried out except that 0.5 g of phenylalanine was used instead of 3 g of ascorbic acid.

15

**Comparative Example G:**

Ascorbic acid (3 g) and phenylalanine (0.5 g) were dissolved in 100 mL hot water. This solution was heated on a boiling water bath for 5 min. The hot solution was sprayed on to 100 grams of black tea. This was incubated at 25 - 30°C for about 15 minutes followed by drying on a fluidised bed drier at 130 - 140°C to bring down the moisture to less than 5% on black tea basis.

The teas thus prepared in Comparative Examples A to G and Example 1 were tested for infusion in water by the following method:

**Process for preparing cold water infusions**

30 1 g of each black tea sample was infused in 100 ml of water at 25°C for 5 minutes. The solutions were filtered to give cold tea infusions and the colour of infusions obtained from Comparative Examples A-G and Example 1 was measured using a

Hunter Lab Ultrascan XE™ colorimeter in the transmittance mode. The data is presented in Table 1.

#### Colour measurements

5

L\*a\*b\* measurements for colour were carried out on Hunterlab UltraScan XE™ colorimeter under the following conditions:

Cuvette - 2 cm (Quartz), Mode - Transmittance, Illuminant -

10 D65, Observer - 10, Scale - CIELAB. The reflectance at 520 nm was also determined.

50 ml of the brew was taken in a 2 cm quartz cuvette, transmittance/reflectance was measured under the conditions 15 mentioned above.

The a\* values are reported as these denote redness of the tea.

The higher the value, the redder the tea. 520 nm is the wavelength at which the red colour absorbs. The higher the 20 absorbance value the greater the red colour.

Table 1:

Example	a*	Absorbance
		520 nm
A	28.6	0.77
B	30.7	0.85
C	21.4	0.59
D	28.3	0.78
E	41.5	1.10
F	29.4	0.83
G	25.0	0.66
1	50.4	1.51

25 Data in Table 1 indicates that there is synergistic benefit in adding ascorbic acid, phenylalanine and hydrogen peroxide to the black tea compared to adding any one or combinations of any two of these ingredients.

**Rate of Infusion**

The rate at which the tea infuses into cold water was also studied. The effect was studied for Comparative Examples A, E and Example 1. The 5 colour of the infusion after 30 seconds, 3 and 5 minutes was determined. The data is presented in Table 2.

**Table 2:**

Example	Time (minutes)	a*	Absorbance (520 nm)
A	0.5	4.31	0.28
	3.0	18.91	0.55
	5.0	23.98	0.66
E	0.5	26.71	0.64
	3.0	30.27	0.73
	5.0	39.06	1.02
1	0.5	31.98	0.72
	3.0	45.24	1.17
	5.0	49.93	1.49

10

The data presented in Table 2 shows that the teas prepared by the process of the invention (Example 1) infuse faster and give better red colour within a short period as compared to control 15 teas or teas treated with ascorbic acid and hydrogen peroxide alone.

It is thus possible by way of the present invention to produce a-black leaf tea that infuses quickly in hot or cold water and 20 provides for a good red colour.

**Aroma/Flavour enhancement - Head Space Gas Chromatography**

The additional benefit of this invention which is aroma / flavour enhancement is demonstrated by measuring the levels of

key pleasant flavoured molecules like Phenyl acetaldehyde and benzaldehyde. The data for the comparative examples from A to G and example 1 are given in Table 3.

5 Table 3:

Example	Phenyl acetaldehyde (Peak area)	Benzaldehyde (Peak area)
A	15349	33937
B	11063	28654
C	9683	42449
D	8522	37137
E	6679	50156
F	153824	77466
G	75130	92055
1	1770134	96077

**Comparative Example H:**

An experiment as per Example 1 was carried out without using

10 black tea and the colour a\* was measured at zero, 30 and 60 minutes in comparison to Example 1 and Comparative Example A.

The data is summarized in Table 4

Table 4:

Example	a* at zero time	a* after 30 minutes	a* after 60 minutes
A	21.79	17.17	15.84
1	50.63	54.27	53.65
H	-0.38	-0.62	-0.79

15

Table 4 demonstrates that black tea is essential for producing the red colour.

It is thus possible by way of the present invention to produce a black leaf tea that infuses quickly in hot or cold water and provides for a good red colour.

Claims

1. A process for the manufacture of a tea product comprising:
  - (i) contacting black tea with ascorbic acid its salts or mixtures thereof, one or more amino acids, an oxidizing agent and water for a period of at least 5 minutes; followed by
  - (ii) drying to prepare a tea product that is infusible in water at 5 to 100°C.
- 10 2. A process according to claim 1 wherein the black tea is contacted with aqueous solutions of the ascorbic acid its salts or mixtures thereof, the one or more amino acids and the oxidising agents.
- 15 3. A process according to claim 1 or claim 2 wherein the ascorbic acid its salts or mixtures thereof is in an amount of 1 to 8% by weight of black tea.
- 20 4. A process according to claim 3 wherein the ascorbic acid its salts or mixtures thereof is in an amount of 2 to 4% by weight of black tea.
- 25 5. A process according to any of claims 1 to 4 wherein the one or more amino acids are in an amount of 0.1 to 8% by weight of black tea.
- 30 6. A process according to claim 5 wherein the one or more amino acids are in an amount of 0.1 to 2% by weight of black tea.
7. A process according to any of claims 1 to 6 wherein the one or more amino acids are chosen from one or more of alanine, arginine, asparagine, cystine, glutamine, glycine, histidine, isoleucine, leucine, lysine, methionine,

phenylalanine, serine, threonine, tryptophan, tyrosine, valine.

8. A process according to any of claims 1 to 7 wherein the  
5 oxidising agent is in an amount of 0.3 to 7% by weight of black tea.
9. A process according to claim 8 wherein the oxidising agent is in an amount of 0.3 to 5% by weight of black tea.  
10
10. A process according to any of claims 1 to 9 wherein the oxidising agent is chosen from one or more of hydrogen peroxide, calcium peroxide, magnesium peroxide, oxygen, an enzyme which can release hydrogen peroxide.  
15
11. A process according to claim 10 wherein the oxidising agent is hydrogen peroxide.
12. A process according to any of claims 1 to 11 wherein the  
20 oxidising agent is added after the addition of the ascorbic acid its salts or mixtures thereof and the one or more amino acids to black tea.
13. A process according to any of claims 1 to 12 wherein the  
25 black tea is contacted with the ascorbic acid its salts or mixtures thereof, the one or more amino acids and the oxidising agent for an incubation period of 5 minutes to 24 hours.
- 30 14. A process according to any of claims 1 to 13 wherein the water is heated to a temperature of 10 to 60°C before contacting with black tea.

15. A process according to any of claims 1 to 14 wherein the black tea is dried to a moisture content of less than 5% after incubation.
- 5 16. A tea product prepared by the process of any one of claims 1 to 15.
17. A tea product obtainable by the process of any one of claims 1 to 15.

ABSTRACT

A process is provided for the manufacture of a tea product which is readily infusible, has improved red colour and good flavour. The process comprises contacting black tea with 5 ascorbic acid and/or its salts, one or more amino acids, an oxidizing agent and water for a period of at least 5 minutes followed by drying to prepare a tea product that is infusible in water at 5 to 100°C.